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This instrumentation grant was used to design and fabricate an intense neutral particle beamline for use in gas-surface collision experiments. The three-fold differentially pumped beamline is being used in conjunction with a new inelastic electron-surface scattering spectrometer in order to extend our studies of surface reactivity and gas-surface collision phenomena into many areas. New capabilities include the ability to examine surface reaction kinetics occurring *on* surfaces (as opposed to monitoring volatile reaction products), encounters between optically excited and translationally selected molecules and surfaces, gaseous condensation, and surface photochemistry. Future surface oxidation studies involving atomic reactants and radicals will also be possible pending construction of appropriate atomic/molecular nozzle beam sources. The instrumentation assembled under the auspices of this grant has directly enhanced research efforts that are DoD supported (AFOSR and ONR).

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# Instrumentation for Surface Reaction Dynamics

DURIP : AFOSR-89-0153

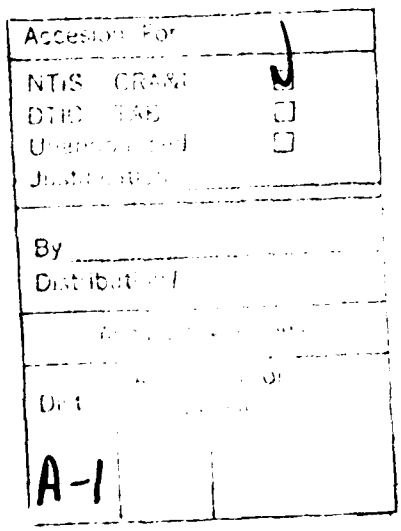
Final Technical Report: December 1, 1988 - November 30, 1989

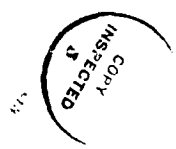
Submitted by Steven J. Sibener
The James Franck Institute and
Department of Chemistry
The University of Chicago
5640 South Ellis Avenue
Chicago, Illinois 60637

## Contents

# ABSTRACT

This instrumentation grant was used to design and fabricate an intense neutral particle beamline for use in gas-surface collision experiments. The three-fold differentially pumped beamline is being used in conjunction with a new inelastic electron-surface scattering spectrometer in order to extend our studies of surface reactivity and gas-surface collision phenomena into many areas. New capabilities include the ability to examine surface reaction kinetics occurring *on* surfaces (as opposed to monitoring volatile reaction products), encounters between optically excited and translationally selected molecules and surfaces, gaseous condensation, and surface photochemistry. Future surface oxidation studies involving atomic reactants and radicals will also be possible pending construction of appropriate atomic/molecular nozzle beam sources. The instrumentation assembled under the auspices of this grant has directly enhanced research efforts that are DoD supported (AFOSR and ONR).



OVERVIEW

During the past year we have designed and fabricated a new differentially pumped neutral particle beamline for use in a variety of gas-surface collision experiments. The new beamline can be used either in conjunction with an inelastic electron-surface scattering spectrometer (primary application) or as a stand-alone unit. The concept involved in a combined molecular beam electron energy loss spectrometer facility (hereafter referred to as a "beam-EELS apparatus) is rather straightforward: a molecular beam of controlled kinetic and internal energy strikes a surface in an ultra-high vacuum environment while an inelastic electron scattering spectrometer is used to concurrently take the *vibrational* or *electronic* spectrum of the same spot on the surface, i.e. the spatial profiles of the molecular beam and electron beam overlap each other on the surface.

The electron spectrometer we have constructed is now fully operational, and is capable of detecting the vibrational frequencies which are present at clean and adsorbate covered surfaces with high sensitivity and energy resolution ($\Delta E \leq 5$ meV). Component testing for the beamline is now underway. Full system assembly and testing should be completed in the spring quarter of 1990. Software development for combined "beam-EELS" data acquisition is also well underway.

The benefits of the new experimental arrangement can best be understood by noting that at the present time our reactive scattering program depends upon our ability to monitor the characteristics of the volatile reaction products which emanate from amorphous or crystalline targets, i.e. we are "blind" in many instances to the sequence of events which occur on the surface prior to desorption. The new arrangement allows us to conduct real-time measurements of surface reaction kinetics on surfaces. It also allows us to study topics of importance to gas-surface interactions which do not generate volatile reaction products such as surface oxidation, gaseous condensation coefficients, and quantum state-selected encounters between vibrationally (or electronically) and translationally selected molecules and surfaces.

Personnel

Our research group currently consists of eight graduate students, two postdoctoral fellows, and an outstanding undergraduate. Listed in order of seniority, these include: Dr. Jeong-Sook Ha, Dr. Thomas Curtiss, Barbara Gans, Suzanne King, Warren Menezes, Daniel Koleske, Glenn Tisdale, Kevin Peterlinz, Michael Stirniman, Errol Sanchez, and Jesus Melendez. Dr. Ha, W. Menezes, G. Tisdale, and B. Gans were involved with various aspects of this beamline project.

Instrumentation

(i) System Design

Both the inelastic electron scattering instrument and the room which houses it were designed with the addition of the new beamline in mind. The spectrometer chamber was intentionally fabricated with several vacuum ports which focus at the same spot as the spectrometer's electron beam. This arrangement can be clearly seen in Figure 1. In addition, the physical layout of the room was planned to contain the combined beam-EELS instrument. Note also that a separate pump room which holds a large Roots pump was already in place when this beamline project was initiated.

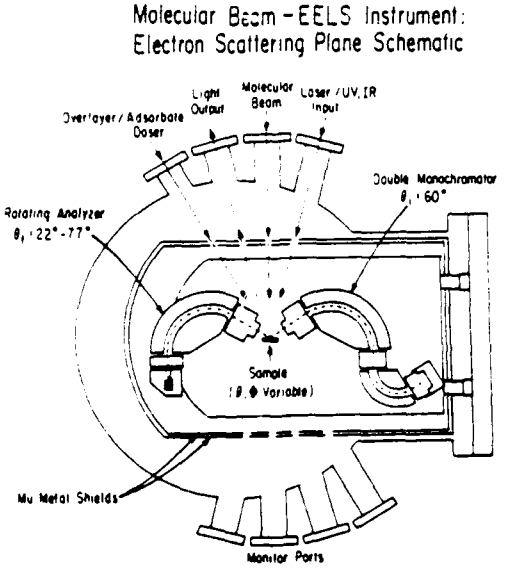



Figure 1: Schematic diagram of the combined molecular beam/inelastic electron scattering apparatus.

Figure 2 shows a side view schematic of the combined "beam-EELS" scattering instrument. The three differential pumping regions of the beamline are clearly visible. The nozzle-skimmer region is on the right, and is pumped by a 4,000 li/sec diffusion pump. The next region contains the beam modulator, and is evacuated by a 1,200 li/sec diffusion pump. The third buffer region, which can also be used for optical pumping if the incident beam, is evacuated by a 240 li/sec turbomolecular pump (chosen for its extreme cleanliness). Finally, the molecular beam enters the ultra-high vacuum electron-surface scattering chamber (left tall chamber in Figure 2).

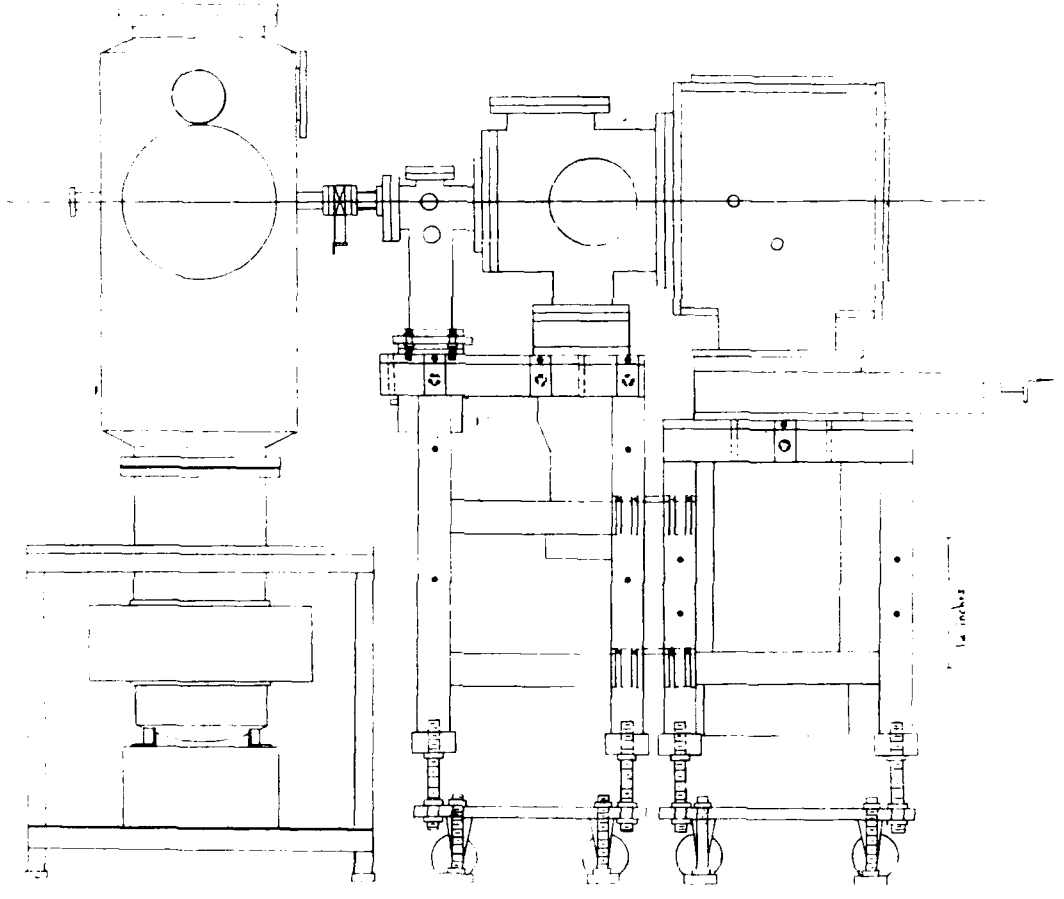


Figure 2: Side view of the combined molecular beam/inelastic electron-surface scattering instrument. The molecular beam originates in the right-most chamber and proceeds towards the left. Three differential pumping regions exist before the molecular beam enters the ultra-high vacuum target chamber.

Finally, Figure 3 shows the internal arrangement of the beamline as it appeared in the original DURIP proposal. Since that time, the outer dimensions of the three differential chambers were modified in order to optimize the performance and flexibility of the final design. (Figure 2 reflects the actual side profiles of these chambers.)

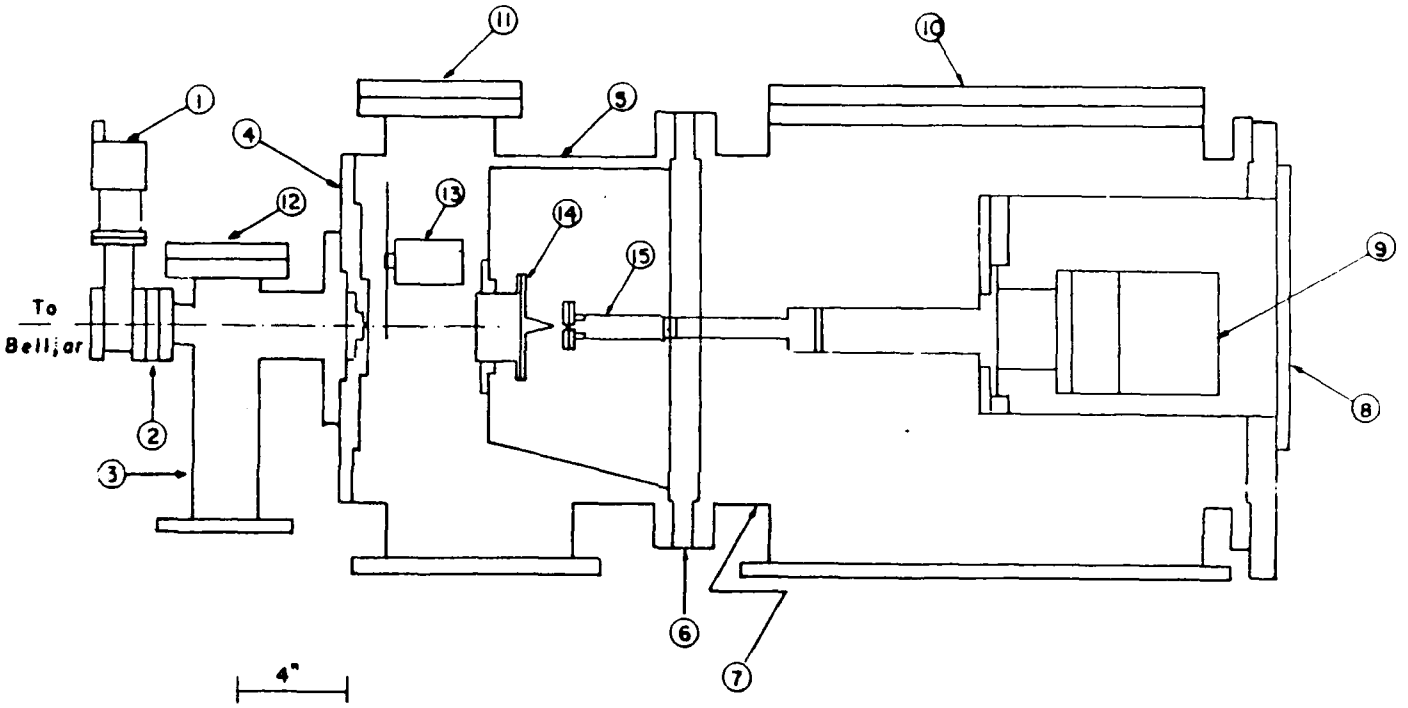



Figure 3: Schematic diagram of the beamline. (1) Manual gate valve (2) Aperture mounting flange (3) Buffer chamber (4) Aperture flange, also for photodiode and Photop (5) Chopper chamber (6) Mating flange (7) Nozzle chamber (8) Beam source mounting flange (9) Molecular beam source (10) Top plate (11) Chopper mounting flange (12) Port for beam intensity measurement (13) Chopper motor (14) Skimmer and its mount (15) Nozzle assembly

(ii) Itemized Beamline Procurement

| | | |
|---|---|---|
| 1. | Beamline construction<br>University of Chicago Central Machine Shop | $ 40,000 |
| 2. | Beamline control electronics<br>University of Chicago - Research Institutes' Electronic Shop | $ 7,101 |
| 3. | Turbomolecular pump package for Region 3 evacuation<br>Balzers Corp: Model TPU240 | $ 6,735 |
| 4. | UHV ionization gauge controller for pressure measurement<br>Perkin-Elmer: Model DGCIII w/options and gauges | $ 3,262 |
| 5. | Mechanical pumps for foreline evacuation<br>Alcatel Corp: Model UM2008A ($1,278); Model UT2020 ($1,809) | $ 3,087 |
| 6. | Miscellaneous Stainless Steel Hardware<br>MDC Corporation ($2,272); Perkin-Elmer ($ 68) | $ 2,340 |
| 7. | Cryocooler for trap refrigeration<br>Neslab: Model CC-100L | $ 2,095 |
| 8. | Low vacuum ionization gauge controller for Regions 1 and 2 pressure measurement<br>Granville-Phillips: Series 270 w/gauges | $ 1,353 |
| 9. | Temperature controller for nozzle regulation<br>Eurotherm: Model 818P w/options. | $ 1,145 |
| 10. | Chopper for beam modulation<br>Stanford Research Systems: Model SR 540 | $ 795 |
| 11. | Hard disk for data handling<br>Cambridge Automation: Model DK515-78 | $ 562 |
| 12. | Linear motion feedthrough for beam chopper positioning<br>Huntington Mech. Labs: Model VF-108-1 | $ 425 |
| | TOTAL | $ 68,900 |

Cost Sharing - University of Chicago

The laboratory that houses the new inelastic electron scattering/molecular beam instrument was completely renovated by the university at a cost in excess of $50,000. This renovation incorporated extensive vacuum line routing (12" diameter) and fabrication of a separate mechanical pump room in anticipation of the beamline's addition to this facility.

Additional costs associated with the prior construction of our inelastic electron scattering spectrometer were covered, in part, by grants from earlier DoD-URIP programs, Dow Chemical, the Research Corporation, the NSF-MRL program, and the Louis Block Fund at the University of Chicago.

Presentations

Invited seminars were presented by Steven J. Sibener at the following locations during the time period of this grant:

Ontario Laser and Lightwave Research Centre
University of Toronto
Toronto, Canada

SPIE OE/LASE '89
Symposium on Photochemistry in Thin Films
Los Angeles, California

National ACS Meeting (April 1989)
Symposium on Diffusion and Reactivity at Surfaces
Dallas, Texas

Gordon Research Conference on Molecular Energy Transfer
Session Chair and Overview Presentation on Gas-Surface Interactions
Wolfboro, New Hampshire

Gordon Research Conference on the Dynamics of Gas-Surface Interactions
Andover, New Hampshire

International Workshop on Surface Dynamics
University of Texas
Austin, Texas

Publications

Our efforts associated with this new DURIP supported program have been exclusively devoted to beamline design, fabrication, and assembly during the past year. Accordingly, no publications have yet resulted from this work.

DoD Consulting

The Principal Investigator is a consultant for the Institute for Defense Analyses, and was a member of the Defense Science Study Group (term now completed).

Summary

We would like to emphasize in closing that the DURIP grant that was awarded certainly achieved its goal of allowing us to construct a novel molecular beam/inelastic electron-surface scattering facility for conducting a variety of surface science experiments. The research being pursued should allow us to address important topics in the area of gas-surface interactions that were previously inaccessible to measurement. The instrumentation assembled under the auspices of this grant has directly enhanced research efforts that are DoD supported (AFOSR and ONR).

# STEVEN J. SIBENER

November 1989

**Born**

April 3, 1954; Brooklyn, New York

**Education**

1971-1975 University of Rochester, Rochester, New York.
Sc.B. in Chemistry awarded with High Distinction, 1975.
B.A. in Physics awarded with Distinction, 1975.

1975-1979 University of California, Berkeley.
M.S. in Chemistry, 1977.
Ph.D. in Chemistry, 1979.
Research with Professor Yuan T. Lee.

**Professional Experience**

Eastman Kodak Research Laboratories, Photographic Research Division, Summer 1974: Electrostatic properties of polymers and polymer-metal interfaces.

Eastman Kodak Research Laboratories, Physics Division, Solid State Physics Research Laboratory, Summer 1975: Oxide growth on GaAsP for MOS fabrication.

Bell Laboratories Postdoctoral Fellow, September 1979 - August 1980. Research with Dr. M.J. Cardillo involving molecular beam scattering from single crystal surfaces.

The University of Chicago, The James Franck Institute and
The Department of Chemistry:
Assistant Professor, August 1980 - June 1985.
Associate Professor, July 1985 - June 1989.
Professor of Chemistry, July 1989 - .

**Honors and Awards**

Marlow Medal of the Faraday Division of the Royal Society of Chemistry, 1988.
IBM Faculty Development Award, 1984-86
Alfred P. Sloan Foundation Research Fellow, 1983-87
Camille and Henry Dreyfus Young Faculty in Chemistry Award, 1980.
Gulf Oil Research Fellow, University of California, Berkeley
American Institute of Chemists Award, University of Rochester
American Chemical Soc. Div. of Colloid and Surface Chemistry Undergraduate Thesis Competition Honorable Mention: "The Shape of Liquid Interfaces," 1975.
Regional Scholar for New York City, University of Rochester

**Associations**

Phi Beta Kappa
American Physical Society
American Chemical Society
Royal Society of Chemistry
Sigma Xi
AAAS

S.J. Sibener **Curriculum Vitae**

**Invited Lectureships**

Physikalisches Institut der Universität Erlangen-Nürnberg, 1988

**Consulting**

| | |
|---|---|
| Dow Chemical USA | (1982-1985) |
| Teltech Resource Network | (1985- ) |
| Institute for Defense Analyses | (1985- ) |

**Professional Activities**

Vice-Chairman, 1985 Gordon Research Conference on the Dynamics of Gas-Surface Interactions

Member, Defense Science Study Group--Institute for Defense Analyses (1985 - 1988).

Member, Materials Research Laboratory Policy Committee, The University of Chicago (1987 - ).

Chairman, 1987 Gordon Conference on the Dynamics of Gas-Surface Interactions.

Member, International Advisory Committee of the Vibrations at Surfaces V Conference (September 1987).

Member, Board of Trustee's Visiting Committee for the College of Arts and Science, University of Rochester (July 1, 1987 - ).

Member, Council of the University Senate, The University of Chicago (September 1989 - September 1992).

Member, IQEC '90 Program Committee (May 1990).